Standard Operating Procedure for Toxicity Characteristic Leaching Procedure (TCLP)

1.0 Location

This procedure is conducted in spectroscopy laboratory, room #305

2.0 Purpose

Toxicity Characteristics Leaching Procedure is used to identify those wastes which are hazardous and thus subject to regulation under subtitle C of the Recourse Conservation and Recovery Act (RCRA) due to their potential to leach significant concentration of specific toxic constituents.

3.0 Scope

This procedure prepares the samples for analysis by GFAA, ICP and mercury by cold vapor AA technique.

4.0 Reference

SW-846 Method 1311.

5.0 Sample Handling & Preservation

5.1 All samples shall be collected using an appropriate sampling plan.

5.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state of the waste and the analysis of concern.

5.3 Preservatives shall not be added to samples before extraction.

5.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs the entire sample should be extracted.

5.5 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction.

5.6 Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH<2 unless precipitation occurs.

6.0 Apparatus and Materials

6.1 Agitation apparatus

6.2 Extraction vessels

6.3 Filter holder

6.4 Filter devices

6.5 pH meter

6.6 Laboratory balances

6.7 500 mL beakers or erlenmeyer flasks

6.8 Watch glasses

6.9 Magnetic stirrer

7.0 Reagents

7.1 Deionized water

7.2 Hydrochloric Acid (1N) HCl made from ACS reagent grade

7.3 Sodium hydroxide (1N) NaOH made from ACS reagent grade

7.4 Glacial Acetic Acid CH_3CH_2COOH, ACS reagent grade

7.5 Extraction fluid

Note: These extraction fluids should be monitered frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications the fluid shall be discarded and fresh extraction fluid prepared.

7.5.1 Extraction fluid #1

Add 11.40 mL glacial acetic acid and 128.6 mL of 1N NaOH to a 2.0 L Volumetric flask and dilute with water to the mark. The pH of this fluid will be 4.93 +/- 0.05.

7.5.2 Extraction fluid #2

Dilute 11.4 mL glacial Acetic Acid with water to a volume of 2.0L. When correctly prepared, the pH of this fluid will be 2.88 +/- 0.05.

8.0 Procedures

8.1 Determination of the per cent solid

8.1.1 If the waste obviously yield no liquid subject to pressure filtration or if visually 100 % solid proceed to ----

8.1.2 If the sample is liquid or multiphase liquid/solid separation to make a preliminary determination of percent solid is required.

8.1.3 Pre weigh the filter and the container that will receive the filtrate.

8.1.4 Weigh out a subsample of the waste and record the weight.

8.1.5 Quantitatively transfer the waste sample to the filter holder. Spread the waste sample evenly over the surface of the filter.

8.1.6 Gradually apply vaccum or gentle pressure of 1 to 10 psi until gas moves through the filter. If no additional liquid has passed through the filter in any 2 minute interval slowly increase the pressure in 10 psi increments to a maximum of 50 psi.

8.1.7 The material in the filter holder is defined as the solid phase of the waste and filtrate is defined as the liquid phase.

8.1.8 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the total waste sample.

8.1.9 Record the weight of the liquid and solid phase. Calculate the percent solids as follows :

Percent of solid = (Wt. of solid /total wt of waste) x 100

8.1.10 If the percent solid determined is equal to or greater than 0.5 % , determine whether the solid material requires particle size reduction. Particle size reduction is required unless the solid has a surface area per gram equal to or greater than 3.1 cm^2.

8.2 Determination of appropriate extraction fluid

8.2.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid to a particle size of approximately 1 mm. Transfer 5.0 g of sample to a 500 mL beaker. Add 96.5 mL of water to the beaker stir 5 minutes using magnetic stirrer. Measure and record the pH. If pH is <5.0, use extraction fluid #1.

8.2.2 If the pH is >5.0 add 3.5 mL 1 N HCl slurry briefly, cover with watch glass heat to 50 C for 10 minutes. Let the solution cool to room temperature and record the pH. If the pH is <5.0 use extraction fluid #1. If the pH is >5.0 use extraction fluid #2. (1 N = 8.3g HCL to a total of 100 ml Water)

8.3 Extraction

8.3.1 If the aliquot of the waste used for the preliminary evaluation was determined to be 100 % solid, it can be used as follows : A minimum sample size of 70g is recommended.

8.3.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves filtration devices.

8.3.3 Pre weigh the container that will receive the filtrate.

8.3.4 Weigh out a subsample and record the weight. If waste contains <0.5 % solid, the liquid portion of the waste is defined as TCLP.

8.3.5 For waste containing >0.5 % dry solids use the percent solids information.

8.3.6 Quantatively transfer the waste sample to the filter holder, spread the waste sample over the surface of the filter.

8.3.7 Gradually apply pressure of 1 to 10 psi untill gas moves through the filter. If no additional liquid has passed through the filter in any 2 minute interval slowly increase the pressure in 10 psi increaments to a maximum of 50 psi and stop filtration.

8.3.8 The material in the filter holder is defined as the solid phase and the filtrate is defined as the liquid phase may now be ready to analyze.

8.3.9 If the waste contains <0.5% solid proceed to

8.3.10 If the waste contains >0.5% solid and if particle size reduction of the solid was needed.

8.3.11 If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material in to an extractor bottle along with the filter used to separate the initial liquid from solid phase. Prepare the solid portion of the waste for extraction by cutting or grinding the waste to a surface area or particle size which passes through 9.5 mm sieve.

8.3.12 Determine the amount of extraction fluid to add to the extractor vessel as follows :
Weight of extraction fluid = 20 X percent solids X weight of waste /100. (For example: 70g sample and 1400ml TCLP fluid.)
Slowly add this amount of appropriate extraction fluid to the extractor vessel. Close the extractor bottle. Secure in rotary agitation device and rotate at 30 rpm for 18+/- 2 hours. Following the 18 +/- 2 hour extraction separate the material in the extractor vessel through filtration.

8.4 Prepare the TCLP extract as follows :

8.4.1 If the waste contained no initial liquid phase the filtered liquid material is defined as the TCLP extract. You must first acid wash the filter by running through 200 ml of water and 13 ml of nitric acid . Discard, run through another 200 ml water, discard. Run through another 200 ml , collecting it in a 200 ml bottle. Label bottle with wash #.

8.4.2 If compatiable (e.g multiple phases will not result on combination) combine the filtered liquid with the initial liquid phase of the waste. This combined liquid is defined as TCLP extract.

8.4.3 If the initial liquid phase of the waste is not compatible with the filtered liquid, do not combine these liquids but combine the analytical results mathematically.

8.4.4 Following collection of TCLP extract the pH, date, sample number, wash # and person who did the extraction should be recorded. Immediately preserve the extract for analysis. Metals must be acidified to pH <2.

8.5 Analyze the TCLP extract

8.5.1 Analyze the TCLP extract according to the appropriate analytical methods.

8.5.2 If the individual phases are to be analyzed separately, determine the volume of the individual phases, conduct the appropriate analysis and combine the results mathematically by using a simple volume weighted average :
Final analyte Concentration = (V1C1+V2C2)/(V1+V2)
Where
V1 = The volume of the first phases (L)
C1 = The concentration of the analyte of concern in the first phase (mg/L)
V2 = The volume of the second phase (L)
C2 = The concentration of the analyte of concern in the second phase (mg/L)

9.0 Quality Assurance

9.1 Maintain all data, including quality assurance data and keep it available for referance or inspection.

9.2 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

9.3 A matrix spike shall be performed for each waste unless the result exceeds the regulatory level. If more than one sample of the same waste is being tested, a matrix spike needs to be performed for every twenty samples and average percent recovery applied to the waste characterization.

9.4 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. The purpose of the matrix spike is to monitor the adequacy of the analytical methods used on the TCLP extract. If the spike recoveries are less than 50%, then the analytical methods are not performing adequately. Use of internal calibration quantitation methods, modification of the analytical methods or use of alternate analytical methods may be needed to accurately measure the contaminant concentration in the TCLP extract. Use of internal quantitation methods is also required when the contaminant concentration is within 20% of the regulatory level. Matrix spike recoveries are calculated by the following formula :

Percent Recovery = {(A-B)/ C} X 100%
Where A =the concentration of the spiked sample, B = the concentration of the unspiked sample and C = the spike level.

9.5 The use of internal calibration quantitation methods shall be employed for a contaminant if

9.5.1 Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and

9.5.2 The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

9.6 The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

9.7 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. The first addition should be prepared so that the resulting concentration is approximately 50 % of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100 % and 150 % of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrumental technique. All four aliquots are analyzed.

9.8 Prepare a plot or subject data to linear regression of instrumental signals or external - calibration - derived concentration as the dependent variable (Y - axis) versus concentrations of additions of standard as the independent variable (X - axis). Solve for the intercept of the abscissa which is the concentration of the unknown.